

PATENT COOPERATION TREATY

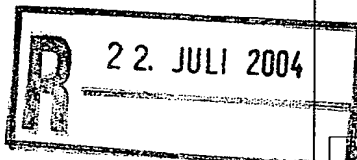
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From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

PCT

To:

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NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL PRELIMINARY
EXAMINATION REPORT
(PCT Rule 71.1)

Date of mailing
(day/month/year)

19.07.2004

Applicant's or agent's file reference
2002FR302

IMPORTANT NOTIFICATION

International application No.
PCT/B 03/03398

International filing date (day/month/year)
28.07.2003

Priority date (day/month/year)
30.07.2002

Applicant
CLARIANT (FRANCE) et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.
4. **REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

The applicant's attention is drawn to Article 33(5), which provides that the criteria of novelty, inventive step and industrial applicability described in Article 33(2) to (4) merely serve the purposes of international preliminary examination and that "any Contracting State may apply additional or different criteria for the purposes of deciding whether, in that State, the claimed inventions is patentable or not" (see also Article 27(5)). Such additional criteria may relate, for example, to exemptions from patentability, requirements for enabling disclosure, clarity and support for the claims.

Name and mailing address of the international preliminary examining authority:



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PATENT COOPERATION TREATY
PCT
INTERNATIONAL PRELIMINARY EXAMINATION REPORT
(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 2002FR302	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/PEA/416)	
International application No. PCT/IB 03/03398	International filing date (<i>day/month/year</i>) 28.07.2003	Priority date (<i>day/month/year</i>) 30.07.2002
International Patent Classification (IPC) or both national classification and IPC C07C209/42		
Applicant CLARIANT (FRANCE) et al.		



1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 4 sheets, including this cover sheet.

☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 10 sheets.

3. This report contains indications relating to the following items:

I	<input checked="" type="checkbox"/>	Basis of the opinion
II	<input type="checkbox"/>	Priority
III	<input type="checkbox"/>	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
IV	<input type="checkbox"/>	Lack of unity of invention
V	<input checked="" type="checkbox"/>	Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
VI	<input type="checkbox"/>	Certain documents cited
VII	<input type="checkbox"/>	Certain defects in the international application
VIII	<input type="checkbox"/>	Certain observations on the international application

Date of submission of the demand 04.02.2004	Date of completion of this report 19.07.2004
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized Officer Jardon Alvarez, J Telephone No. +49 89 2399-8325 

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/IB 03/03398

I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, Pages

1-45 as originally filed

Claims, Numbers

1-16 filed with telefax on 17.06.2004

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).
3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:
- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:
5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)).

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/B 03/03398

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability;
citations and explanations supporting such statement**

1. Statement

Novelty (N)	Yes: Claims	1-16
	No: Claims	
Inventive step (IS)	Yes: Claims	1-16
	No: Claims	
Industrial applicability (IA)	Yes: Claims	1-16
	No: Claims	

2. Citations and explanations

see separate sheet

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

D1: Katritzky A. R. et al., Tetrahedron: Asymmetry, 7, (6), 1996, 1621-1630

1. None of the available prior art documents discloses a process for the preparation of primary amines by reaction of a triazolium salt with a hydride as claimed in claims 1 to 10, 13 and 14 or compounds as claimed in claims 11, 12, 15 and 16. The subject-matter of the claims is therefore novel (Article 33(2) PCT).
2. The subject-matter of the claims also involves an inventive step (Article 33(3) PCT).
 - 2.1. The closest prior art document, D1, discloses the use of (S,S)-4-amino-3,5-bis(1-hydroxyethyl)-1,2,4-triazole as chiral auxiliary in diastereoselective alkylation reaction on CN double bonds of hydrazones (see D1, Scheme 1). However, D1 does not disclose the removal of this chiral auxiliary for the preparation of amines (see page 1625, lines 17 - 18).
 - 2.2. The problem underlying the present application can then be seen as to find a process for the preparation of primary amines using the 4-amino-1,2,4-triazole synthons.

This problem is solved by the process of claim 1 by using a triazolium salt of formula (II) which is treated with a hydride. The process allows the preparation of the desired amines in good yields (see examples 1 and 2). There is no hint to this solution into the available prior art and therefore an inventive step is acknowledged for the process of claim 1 (Article 33(3) PCT).
 - 2.3. The subject-matter of dependent claims 2 to 10, 13 and 14 which includes the inventive feature of claim 1 also involves an inventive step as required by Article 33(3) PCT.
 - 2.4. The subject-matter of claims 11, 12, 15 and 16 which is directed to novel intermediates in the inventive process of claim 1 also involves an inventive step due to its contribution to the inventive process of claim 1 (Article 33(3) PCT).

CLAIMS

- 1. Process for the preparation of primary amines of formula (I):**

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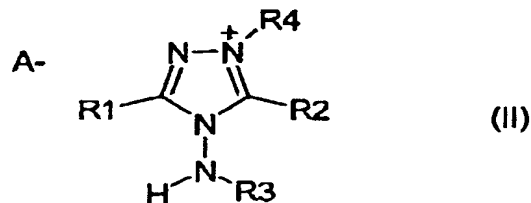
in which

R3 represents

- 10 - a linear or branched alkyl group including from 1 to 6 carbon atoms which is optionally substituted by one or more hydroxyl groups, amino groups, alkoxy groups including from 1 to 6 carbon atoms or aryl groups including from 6 to 10 carbon atoms, the aryl groups optionally being substituted by one or more linear or branched alkyl groups including from 1 to 6 carbon atoms or by one or more alkoxy groups including from 1 to 6 carbon atoms or by one or more phenyl groups,
- 15 - a cycloalkyl group including from 5 to 7 carbon atoms which is optionally substituted by one or more linear or branched alkyl groups including from 1 to 6 carbon atoms by or one or more alkoxy groups including from 1 to 6 carbon atoms,
- 20 - an aralkyl group including from 7 to 16 carbon atoms which is optionally substituted by one or more linear or branched alkyl groups including from 1 to 6 carbon atoms, by one or more alkoxy groups including from 1 to 6 carbon atoms or by one or more phenyl groups,

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by reaction of a triazolium salt of formula (II):



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in which:

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R1 and R2, which are identical or different, represent

- hydrogen,
- a linear or branched alkyl group including from 1 to 6 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms, $-(OCH_2CH_2O)_nR'''$ groups in which n represents an integer ranging from 1 to 4 and R''' is a linear or branched alkyl group including from 1 to 4 carbon atoms, -O-aryl groups including from 6 to 10 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms, linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups, or -O-aralkyl groups including from 7 to 16 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms, linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups;
- an aralkyl group including from 7 to 16 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms, linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups;
- an aryl group including from 6 to 10 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups,

R3 has the meaning already indicated,

R4 represents

- a linear or branched alkyl group including from 1 to 6 carbon atoms which is optionally substituted by a -COOH radical or a -COOR''' group in which R''' represents a linear or branched alkyl radical including from 1 to 4 carbon atoms,
- an aralkyl group including from 7 to 16 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms or a -COOH radical or a -COOR''' group in which R''' represents a linear or branched alkyl radical including from 1 to 4 carbon atoms,
- a residue of an organic polymer functionalized by an alkylating group,

A represents

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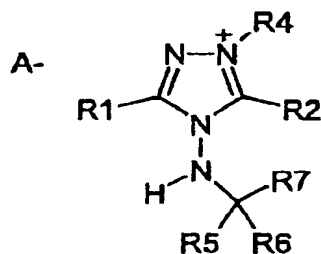
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- a halogen,
- an alkylsulphonate group including from 1 to 6 carbon atoms which is optionally substituted by one or more halogen groups,
- an arylsulphonate group including from 6 to 10 carbon atoms which is optionally substituted by one or more halogen groups or linear or branched alkyl groups including from 1 to 4 carbon atoms,
- an alkyl sulphate group including from 1 to 6 carbon atoms,
- a hydrogen sulphate group,
- a hemisulphate group,
- a perchlorate group, or
- a hydroxide group.

with a hydride, to obtain the amine of formula (I), which is isolated, if desired.

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2. Process according to Claim 1, characterized in that the R3 group comprises an asymmetric carbon α to the nitrogen.
3. Process according to Claim 1 or 2, characterized in that the triazolium salt of formula (II) corresponds to the formula (IIa):



(IIa)

in which

- 25 R1, R2, R4 and A have the meaning already indicated and R5 represents

- a hydrogen,
- a linear or branched alkyl group including from 1 to 6 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms, hydroxyl groups or amino groups,

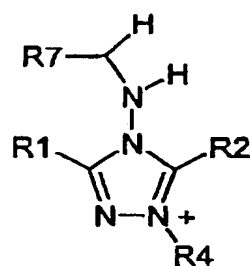
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- a cycloalkyl group including from 3 to 7 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms,
- 5 - an aryl group including from 6 to 10 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups,
- 10 - an aralkyl group including from 7 to 16 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms, linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups,

R6 represents

- 15 - a linear or branched alkyl group including from 1 to 6 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms, hydroxyl groups or amino groups,
- a cycloalkyl group including from 3 to 7 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms,
- 20 - an aryl group including from 6 to 10 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups,
- 25 - an aralkyl group including from 7 to 16 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms, linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups,
- 30 - an aminotriazolium group of formula

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A-

(IIb)

in which R1, R2, R4 and A have the meaning already indicated,

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R7 represents

- a hydrogen,
- a linear or branched alkyl group including from 1 to 6 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms, hydroxyl groups or amino groups,
- a cycloalkyl group including from 3 to 7 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms,
- an aryl group including from 6 to 10 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups,
- an aralkyl group including from 7 to 16 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups, or

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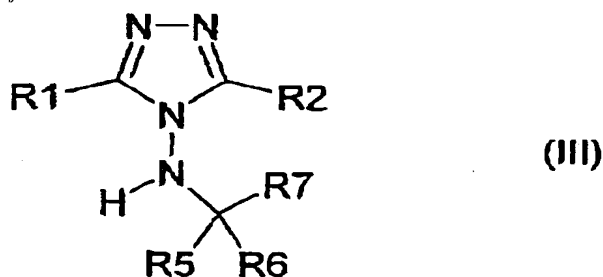
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R5 and R6 can form, together with the carbon atom to which they are bonded, a ring comprising 5 to 7 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms,

it being understood that the carbon carrying the R5, R6 and R7 radicals must be asymmetric.

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4. Process according to one of Claims 1 to 3, characterized in that the compound of formula (II) corresponds to the formula (IIa) defined above in Claim 3 and, in addition, the said compound of formula (IIa) is prepared by reaction of a compound of formula (III):

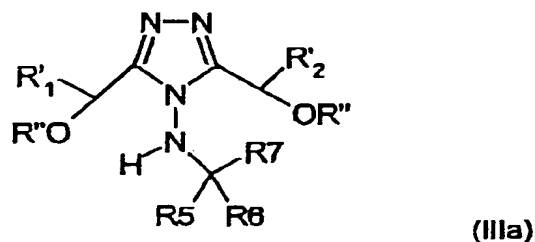


in which

R1, R2, R5, R6 and R7 have the meaning already indicated in Claim 3,

with an agent for the quaternization of a nitrogen, to produce the compound of formula (IIa), which is isolated, if desired, or which is employed directly in the following stage.

5. Process according to one of Claims 1 to 4, characterized in that the compound of formula (II) corresponds to the formula (IIa) defined above in Claim 3 and, in addition, the said compound of formula (IIa) is prepared by reaction of a compound of formula (IIIa):



in which

R5, R6 and R7 have the meaning already indicated in Claim 3,

R'1 and R'2 represent

- 5
- a linear or branched alkyl group including from 1 to 6 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms,
 - an aryl group including from 6 to 10 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups, or
 - an aralkyl group including from 7 to 16 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups, and
- 10

R" represents

- 15
- hydrogen,
 - a linear or branched alkyl group including from 1 to 6 carbon atoms,
 - an aryl group including from 6 to 10 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups,
- 20
- an aralkyl group including from 7 to 16 carbon atoms which is optionally substituted by one or more alkoxy groups including from 1 to 6 carbon atoms or linear or branched alkyl groups including from 1 to 6 carbon atoms or phenyl groups,
 - a $-(CH_2CH_2O)_nR'''$ group in which n represents an integer ranging from 1 to 4 and R''' is a linear or branched alkyl group including from 1 to 4 carbon atoms,
- 25

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with an agent for the quaternization of a nitrogen, to produce the compound of formula (IIa), which is isolated, if desired, or which is employed directly in the following stage.

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6. Process according to Claim 5, characterized in that the compound of formula (IIIa) is additionally prepared by reaction of an organometallic compound of formula

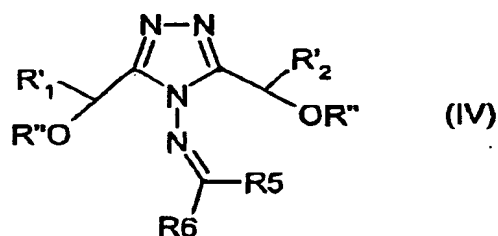
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R7-M

in which R7 has the meaning already indicated in Claim 3 and M represents an MgX or CaX₂ group in which X represents a halogen atom and M represents a metal, such as Li, Cu or (1/2) Zn,

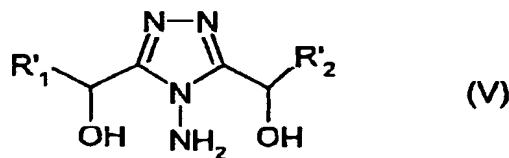
with a compound of formula (IV)



in which R'1, R'2 and R'' have the meaning already indicated in Claim 4 and R5 and R6 have the meaning already indicated in Claim 3, it being understood that, when R'' is a hydrogen, at least one of R5 and R6 is an optionally substituted aryl group,

to produce the compound of formula (IIIa), which is isolated, if desired, or which is employed directly in the following stage.

7. Process according to Claim 6, characterized in that the compound of formula (IV) is prepared by etherification and reaction of a compound of formula (V):



in which R'1 and R'2 have the meaning already indicated in Claim 4, with a compound of formula

O=CR5R6

in which R5 and R6 have the meaning already indicated in Claim 3,

to produce the compound of formula (IV), which is isolated, if desired, or which is employed directly in the following stage.

8. Process according to Claim 7, characterized in that the etherification takes place before the reaction of the compound of formula (V) with the compound of formula $O=CR_5R_6$.

9. Process according to Claim 7, characterized in that the etherification takes place after the reaction of the compound of formula (V) with the compound of formula $O=CR_5R_6$, it being understood that at least one of R5 and R6 represents an optionally substituted aryl group.

10. Process according to Claim 5, characterized in that the compound of formula (IIIa) is additionally prepared by reduction by the action of a metal hydride on a compound of formula (IV) defined above in Claim 6 or by hydrogenation of the said compound of formula (IV), it being understood that R5 cannot, in this case, represent hydrogen.

11. As novel intermediates for preparing an amine of formula $H_2N-CHR_6R_7$, the following compounds:

- 4-[(R)-1-Ethyl-2,2-dimethoxyethylamino]-(S,S)-3,5-bis(1-methoxyethyl)-1,2,4-triazole
- 4-[(S)-1-Ethyl-2,2-dimethoxyethylamino]-(S,S)-3,5-bis(1-ethoxyethyl)-1,2,4-triazole
- 4-[(R)-1-Ethyl-2,2-dimethoxyethylamino]-(S,S)-3,5-bis(1-ethoxyethyl)-1,2,4-triazole
- 4-(1-Phenyl-2,2-dimethoxyethylamino)-(S,S)-3,5-bis(1-methoxyethyl)-1,2,4-triazole
- 4-(1-Ethyl-2,2-dimethoxyethylamino)-(S,S)-3,5-bis(1-(2-methoxyethyl)ethyl)-1,2,4-triazole
- 4-(1-Ethylbutylamino)-(S,S)-3,5-bis(1-methoxyethyl)-1,2,4-triazole

- 4-(1-Ethylisobutylamino)-(S,S)-3,5-bis(1-methoxyethyl)-1,2,4-triazole
- 4-(1-Phenylpropylamino)-(S,S)-3,5-bis(1-methoxyethyl)-1,2,4-triazole
- 4-(1-Phenylethylamino)-(S,S)-3,5-bis(1-methoxyethyl)-1,2,4-triazole
- (Hexyl-3,4-diamino)-4,4'-bis[(S,S)-3,5-bis(1-methoxyethyl)-1,2,4-triazole].

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12. As novel intermediates for preparing an amine of formula $H_2N-CR_5R_6R_7$, the following compounds:

- N-[(S,S)-3,5-Bis(1-methoxyethyl)-1,2,4-triazol-4-yl]-2,2-dimethoxyethylimine
- 10 - N-[(S,S)-3,5-Bis(1-methoxyethyl)-1,2,4-triazol-4-yl]butylimine
- N-[(S,S)-3,5-Bis(1-methoxyethyl)-1,2,4-triazol-4-yl]isobutylimine
- N-[(S,S)-3,5-Bis(1-methoxyethyl)-1,2,4-triazol-4-yl]-1-(ethoxycarbonyl)methylimine
- N-[(S,S)-3,5-Bis(1-methoxyethyl)-1,2,4-triazol-4-yl]-1-phenylethylimine
- 15 - N-[(S,S)-3,5-Bis(1-methoxyethyl)-1,2,4-triazol-4-yl]-1-methyl-2,2-dimethoxyethylimine
- Bis[N-[(S,S)-3,5-bis(1-methoxyethyl)-1,2,4-triazol-4-yl]methylimine]
- N-[(S,S)-3,5-Bis(1-methoxyethyl)-1,2,4-triazol-4-yl]-2,2-dimethoxyethylimine
- N-[(S,S)-3,5-Bis(1-(2-methoxyethoxy)ethyl)-1,2,4-triazol-4-yl]-2,2-dimethoxyethylimine.
- 20

13. Process according to one of Claims 4 to 6, characterized in that, in addition, the stereoisomers of formula (III) or (IIIa) are separated by, optionally chiral, high performance liquid chromatography.

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14. Process according to either of Claims 3 and 5, characterized in that, in addition, the diastereoisomers of formula (IIa) are separated by crystallization.

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15. An enantiomerically pure diastereoisomer of a compound of formula (IIa) obtained according to the process of Claim 14.

35 16. Enantiomerically pure 1-benzyl-4-[(R)-1-phenyl-2,2-dimethoxyethylamino]-(S,S)-3,5-bis(1-methoxyethyl)-1,2,4-triazolium bromide according to claim 15.

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